Publication

CHEMICAL SOURCING OF A PREHISTORIC FRESHWATER SHELL ARTIFACT USING LASER ABLATION-INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

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Three "spoons" made of freshwater mussel (Unionidae) shell were recovered decades ago from a prehistoric burial at Lyon's Bluff, a site in eastern Mississippi located on a tributary stream high up the Tombigbee River drainage. Based on shell morphology, at least one of these artifacts was considered a likely import from the Tombigbee River valley proper. Chemical testing of shell from Lyon's Bluff and sites along the Tombigbee River using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry indicates that the suspected import was fashioned from locally available shell and therefore not imported.

INTRODUCTION

Sourcing artifacts via chemical analysis is a practice that has greatly increased in archaeology in recent years. Artifacts typically are chosen for analysis based on stylistic attributes (e.g., "local" ceramic plainwares vs. stylistically "exotic" sherds), with an assumption that rare types are more likely to be imports (e.g., Herrera et al. 1999; Morrow et al. 2005; Shergur et al. 2003). An analogous argument could be made for what are sometimes called ecofacts, the remains of once-living organisms recovered from archaeological contexts. Such remains may display phenotypic differences related to different source areas (habitats) for the organisms represented, with rare examples of particular phenotypes being considered imports from a non-local environment. In certain situations, such a supposition can be framed as a hypothesis testable via chemical analysis. Here, we describe a freshwater mussel shell that is phenotypically unique at the site from which it was recovered. This shell is a burial accompaniment and has been culturally modified, which, in conjunction with its unique morphology, suggests that it may have been imported to the site. The probable source location based on phenotype is known, allowing direct testing of the hypothesis via chemical analysis. We employ Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) to conduct this test.

FRESHWATER MUSSEL PHENOTYPES AND AQUATIC ENVIRONMENTS

Freshwater mussels (Bivalvia: Unionidae) are known to be phenotypically plastic. Shell size, shape, and sculpture vary with location along a stream, reflecting the expression of particular phenotypes related to substrate composition, current, water depth, and other environmental parameters (Ball 1922; Eager 1978; Ortmann 1920; Roper and Hickey 1994). These changes tend to be clinal; for example, mussels typically grow more obese (greater shell width relative to height) with downstream distance (Claassen 1998; Imlay 1982; Tevesz and Carter 1980). Biologists traditionally have dealt with this phenotypic variability by designating subspecies based on shell morphology, a practice that has been supported by recent genetic testing (e.g., Stiven and Alderman 1992). Theoretically, shell morphologies reflecting different habitats may be employed as sourcing information in archaeological cases.

SHELL FROM THE LYON'S BLUFF SITE

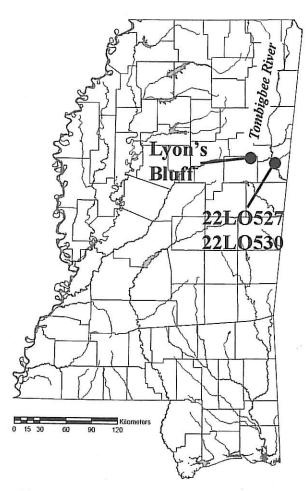


Figure 1. Location of Lyon's Bluff (22OK520) and sites 22LO527 and 22LO530.

Lyon's Bluff (22OK520) is a late prehistoric to early historic period (ca. A.D. 1200 – 1700) mound and village complex located in the Black Prairie physiographic province of eastern Mississippi, U.S.A. (Figure 1). The site is situated beside Line Creek, a tributary of Tibbee Creek, itself a major tributary of the Tombigbee River, the higher-order waterway in the drainage. Lyon's Bluff has been the focus of intermittent archaeological work for decades (Peacock and Hogue 2005). Excavations in 2001 and 2003 produced 920 identifiable freshwater mussel valves representing a minimum of 29 taxa (Peacock et al. 2008). By far the most abundant species is Lampsilis straminea straminea (n = 299. 32.5% of the total). This distinct subspecies is a "small river" form that is smaller, less inflated, and much more highly sculptured with surface ridges than the "big river" form, Lampsilis straminea claibornensis, which is found today along the main stem of the Tombigbee River (Williams et al. 2008) where it also is present in archaeological assemblages (Peacock 2000; Peacock et al. 2008). As can be seen in Figure 2, the two forms are quite distinct.

In 1967, a prehistoric grave (Burial 67-12) at Lyon's Bluff was excavated by Richard Marshall (notes on file, Cobb Institute of Archaeology, Mississippi State University). Accompanying the primary, semi-flexed skeleton of a 7 to 11-year old (unsexed) individual were several artifacts, including what were described in the field notes as three shell "spoons" (O'Hear and Hogue 1995; Elmore 2008). Marshall (1985:62) provides the following description of the burial in question and others recorded at the same time:

Another burial had a number of small shell spoons scattered over it. Still another had a large mussel shell placed on the pelvis which had red pigment adhering to it. Covering the hand of the same burial was a large cut shell spoon of the type common to the Moundville and Duck River Phases.

Elmore (2008) systematically reviewed Marshall's notes, photographs, and other field records from the site. She notes that Burial 67-12 was found in Marshall's Unit 100N20E, Level 6 (Marshall dug in arbitrary six inch levels), and that:

Artifacts associated include: two modified mussel shells...an unfinished shell ornament...one shell spoon...a pottery bowl...two pottery vessels (jars)...one located at the feet of the burial and one at the left shoulder...a raccoon baculum...and cut mica fragments" (Elmore 2008:59).

Although artifacts from burial contexts are relatively rare at Lyon's Bluff (Elmore 2008), it is clear from these descriptions that, when they do occur, artifacts fashioned from freshwater mussel shell are commonly present, along with other materials that are clear imports (e.g., sheet mica, which does not naturally occur in Mississippi).

Mussel shell spoons are a common artifact of the Mississippian period (A.D. 1000 - 1540), being recovered almost exclusively as burial accompaniments. They are usually made from the shells of thinwalled, inflated species such as the Pocketbook, Lampsilis ovata. A handle often is carved into the anterior margin of the shell; this may be plain or may be carved in elaborate forms such as the head of a woodpecker (e.g., Parmalee and Bogan 1998:Fig. 11). Shell spoons are particularly common in the Tennessee and Cumberland River drainages of Tennessee (e.g., Lewis and Lewis 1995; Moore 2004; Thruston 1890; Walling et al. 2000), but specimens have been reported from elsewhere in the eastern United States (e.g., Emerson et al. 2002; Griffin and Morse 1961; Parmalee et al. 1972).

One of the purported spoons from Lyon's Bluff is a left valve of *Lampsilis ornata*, a species that is present, albeit rare, in the total mussel shell assemblage recovered from the site (n = 3). It is an inflated shell showing modification along the anterior margin consistent with other shell spoons from the region

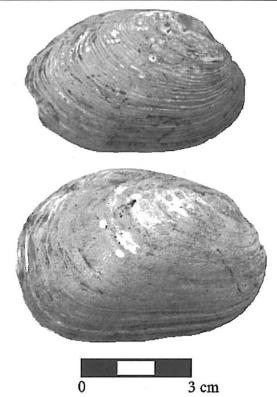


Figure 2. Right valves of *Lampsilis straminea* straminea, the upstream phenotype, and *Lampsilis straminea* claibornensis, the downstream phenotype, from Lyon's Bluff. The lower specimen has been ground along the anterior margin. From Peacock and Jenkins (2010:Fig. 6).

(Elmore 2008:Fig. 11). Another shell is a right valve of *Megalonaias nervosa* which has been lightly ground along the anterior margin; this species also is present, but rare, in the Lyon's Bluff shell assemblage (n = 5). Both *L. ornata* and *M. nervosa* are found today in the Tombigbee River and its tributaries, and they occur in low numbers in archaeological shell assemblages from Tombigbee River sites (Peacock 2000). The third specimen, a right valve of *L. straminea claibornensis*, is not a typical "spoon" form; there is no handle, and the valve is not particularly inflated (Figure 2). The anterior edge has been heavily ground into a smooth arc in a manner similar to, but more pronounced than, the *M. nervosa* specimen. This type of wear is commonly assumed to result from use of a shell as a pottery-smoothing tool (e.g., Parmalee 1988:173-174). While the exact function of this artifact is unknown, its presence in a grave and the fact that it is the only specimen of the morphologically distinct subspecies recovered from the site suggests that it, and possibly the other freshwater shell artifacts associated with Burial 67-12, came from a waterway other than Line Creek. Based on shell morphology, the most likely source for the *L. straminea claibornensis* specimen is further down the drainage in the main stem of the Tombigbee River. The nearest straight-line distance between the Lyon's Bluff site and the Tombigbee River is approximately 25 km (Fig. 1).

METHODS

To test the hypothesis that the *L. straminea claibornensis* shell from Burial 67-12 at Lyon's Bluff is an import, shells from Lyon's Bluff and from two archaeological sites on the main stem of the Tombigbee River

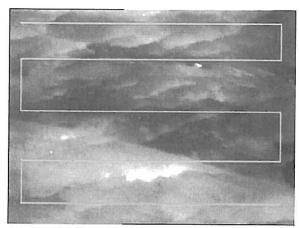


Figure 3. View of sectioned mussel valve showing seasonal growth rings (dark and light bands) and raster pattern set for the laser path. Magnification is 480x.

were subjected to chemical analysis using LA-ICP-MS. From Lyon's Bluff, the L. straminea claibornensis specimen from Burial 67-12 was tested, as were several specimens of L. straminea straminea from Unit 0N20W, Zone F, Level 3, an ashy layer located near the mound (see Peacock and Hogue 2005 for a site plan and unit locations). Site 22LO530, the Shell Bluff site, is a mostly Late Woodland period (ca. A.D. 600 - 900) habitation site (Futato 1987) located on the Tombigbee River. Shell used in this project was collected from eroding midden deposits at the site by personnel from the Mississippi Department of Wildlife, Fisheries and Parks some decades ago, prior to acquisition of the land and alteration of the river by the U.S. Army Corps of Site 22LO527 also is mostly a Late Engineers. Woodland-period, long-term habitation site located near the west bank of the Tombigbee River, approximately

3.5 km south of the Shell Bluff site. Shells from 22LO527 used in this research were obtained from an eroding midden layer or "slump" by Mississippi State University archaeologists in the summer of 1994. We are assuming that shells obtained from non-burial contexts at all sites were locally obtained for mussel consumption (Peacock 2000).

LA-ICP-MS is a high precision method for the chemical characterization of a range of materials (see papers in Speakman and Neff 2005). A laser is used to ablate material, which is then transported from the ablation cell to an ICP torch using an argon gas carrier. The high temperature of the ICP-MS torch ionizes the material before it passes through skimmer cones and into the mass spectrometer where the ions are separated according to their mass/charge ratio. The Perkin-Elmer SCIEX ELAN DRC II used for this research allows for easily resolved peaks and therefore low probabilities of interferences from similar isotopic and molecular species. A computer collects the mass spectra and calculates integrated count rates for the masses of interest.

In total, 28 shells were analyzed, 8 from Lyon's Bluff and ten each from 22LO530 and 22LO527. A number of species from the latter two sites were analyzed to see the extent to which inter-species chemical differences might be a factor in the chemical sourcing of freshwater shell: these include *Elliptio crassidens*, *Epioblasma penita*, *Fusconaia cerina*, *Fusconaia ebena*, *Lampsilis straminea claibornensis*, *Obliquaria reflexa*, *Obovaria* sp. (*unicolor* and/or *jacksoniana*), *Pleurobema decisum*, *Quadrula asperata*, and *Quadrula metanevra*. Size matching was used to insure that no individual animal was represented twice. Shells were first cleaned with deionized, demineralized water, following which they were cut with a diamond-bladed band saw. About two-thirds of the posterior portion of the shell was removed; this was cut in half lengthwise, with the bottom half being retained as a chemical voucher specimen. A 1-cm-wide slice was then removed from the top half for chemical analysis. The saw was cleaned with deionized, demineralized water between samples. Individual slices of shell were mounted in HandiTak® adhesive with a cut side facing up. The *L. straminea claibornensis* shell from Burial 67-12 at Lyon's Bluff was slightly too large to fit in the vacuum chamber, so a small piece was cut from the anterior-dorsal margin and used for testing.

On all shells, a raster pattern was ablated using a New Wave Research UP-213 Laser Ablation system with a 213nm Nd:YAG laser operating at 50% power. A pre-ablation pass was made to remove any adhering surface materials and to allow time for the ICP-MS torch to stabilize after the ablated material is introduced into the plasma. From the midden samples, shells of different sizes were used to average out differences in chemical uptake related to faster growth in younger individuals. The raster pattern covered seasonal growth rings (Figure 3), as there can be seasonal variation in chemical uptake by mollusks. Each specimen was ablated three times, at different locations, and the results averaged.

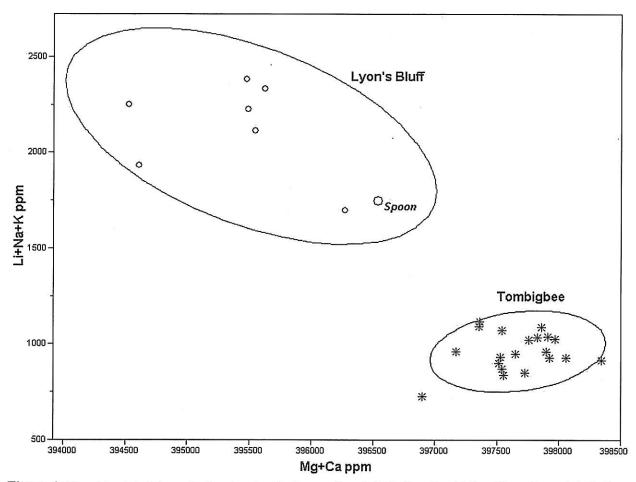


Figure 4. Bivariate plot of elements showing chemical separation of shells from Tombigbee River sites and shells from Lyon's Bluff. Ellipses represent 90 percent confidence intervals.

Data were taken for 46 elements: Li 7, Na 23, Mg 24, Al 27, Si 30, K 39, Ca 44, Sc 45, Ti 47, V 51, Cr 52; Mn 55, Fe 57, Co 59, Ni 60, Cu 65, Zn 66, As 75, Rb 85, Sr 88, Y 89, Zr 90, Nb 93, Sn 120, Sb 121, Cs 133, Ba 138, La 139, Ce 140, Pr 141, Nd 142, Sm 152, Eu 153, Gd 158, Tb 159, Dy 164, Ho 165, Er 166, Tm 169, Yb 174, Lu 175, Hf 180, Ta 181, Pb 208, Th 232, and U 238. Standard materials used for comparison were NIST brick clay SRM 679 and glasses SRM 610 and SRM 612 as well as glasses designated B, C, and D (Brill 1999). Calcium was used as an internal standard. Laser frequency was set at 20Hz. Laser beam diameter was 40 micrometers. Laser speed was set to 70 micrometers/ minute. Plasma gas flow was 17.0 liters/minute, nebulizer gas flow was 1.4 liters/minute, and auxiliary gas flow was 1.2 liters/minute. RF power was 1400W. Analytical time per run was 1 minute; dwell time was 10ms/point. Mass settling time was 3 seconds. Analytical mode was set to full peak scanning mode.

RESULTS

Of the 46 elements examined, a small number served as good discriminators between the Lyon's Bluff and Tombigbee River shell assemblages. The rest either returned similar values or were too near detection limits to be of use. The full corpus of data is available upon request from the senior authors.

Those elements providing the best discrimination were combined for simplicity of presentation and to minimize variation that might arise from different diagenetic environments (Claassen 1998). A bivariate plot of Mg+Ca vs. Li+Na+K (Figure 4) shows good separation between the Tombigbee River sites and the Lyon's Bluff shell assemblage. The *L. straminea claibornensis* from Burial 67-12 falls in with the other shell from Lyon's Bluff, although its relative nearness to the main-stem site assemblages is interesting, and suggests that it may have come from farther downstream than the majority of the shells tested at the site. The shell from the two main-stem central Tombigbee River sites is chemically very similar, indicating that location in the drainage, rather than diagenetic changes (which should vary from site to site), is the main factor structuring the chemical makeup of the shell. While we plan to analyze more shell, with a greater range of species, from Lyon's Bluff in the future, and while diagenetic studies investigation structural changes to the shell are underway, these results are encouraging in that chemical differences between shells from the tributary stream and the main river are great enough to allow clear differentiation using the methods employed.

CONCLUSIONS

Based on shell morphology, it was hypothesized that a culturally modified right valve of *L. straminea claibornensis* found as a burial accompaniment at the Lyon's Bluff site was an import from the main river valley, at least 25 km away, with the implication that other shell "spoons" from the site also were imports. Chemical testing of shell via LA-ICP-MS falsifies this hypothesis, as the burial shell considered the most likely import clearly groups with shells obtained from other deposits at the site, which are chemically distinct from downriver shell assemblages. The subspecies *L. straminea claibornensis* apparently was present, if rare, in Line Creek in prehistoric times. It has been argued that cultural bias in the selection of species was minimal to non-existent in shell from general midden deposits and feature fills in the study area (Peacock 2000). The presence of the single recovered species of *L. straminea claibornensis* in a burial context, coupled with cultural modification of the shell, does suggest that cultural bias was in play at Lyon's Bluff in this particular case.

Although some work has been done on sourcing marine shell artifacts (e.g., Claassen and Sigmann 1993), freshwater mussel shell has received almost no attention in this regard. This is despite the fact that shell import/export has been explicitly hypothesized in particular cases. For example, Theler (1991) suggests that heavy-shelled species of freshwater mussel at the Aztalan site in Wisconsin represent imports from the Mississippi River valley to the west, for use as shell hoes. As we have demonstrated here, such a hypothesis should be readily testable through chemical analysis.

To our knowledge, this is the first time a mussel shell "spoon" has been chemically analyzed. Given that such artifacts are widely found throughout eastern North America, and given their usual context as burial accompaniments, further sourcing studies are warranted. Although bulk analytical methods for chemically analyzing freshwater shell have proven effective (Peacock et al. 2007), we used LA-ICP-MS as part of a continuing study of the chemical content of shell temper in prehistoric ceramics. The high precision obtainable with LA-ICP-MS has led to its use in the study of modern shellfish (e.g., Lorrain et al. 2003; Raith et al. 1996; Takesue and van Geen 2001), and such precision also makes it useful for sourcing archaeological shell. As demonstrated herein, hypotheses concerning artifact origins based on shell morphology can be tested using LA-ICP-MS, and other shell artifacts (e.g., shell beads or gorgets) small enough to fit into the vacuum chamber (5 x 5 x 5 cm) can be tested in a virtually non-destructive manner. Such advantages should lead to an increased use of LA-ICP-MS in analyzing archaeological shell in eastern North America and elsewhere.

End Note

¹The burials at Lyon's Bluff have been determined to be culturally unaffiliated (O'Hear and Hogue 1995).

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